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EXAMINATION OF SAMPLE QUALITY PREPARING BY A.C. CONDUCTIVITY MEASUREMENT

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ABSTRACT: Conductivity measurement of rubber is very sensitive method for evaluation of sample quality. The main problem is relatively low dispersion of fillers, look like carbon black or SiO_2.
This works deals with using of programmable automatic RCL meter – PM 6306 and consequently by measurement of electrical parameters as capacity, impedance, resistance, phase angle, dissipation factor, quality factor by various frequencies.
KEY WORDS: RCL meter, rubber, electrical properties, fillers

1. INTRODUCTION

During the tire products manufacturing the tires or products must verify good driven properties, dimensional stability, minimal rolling resistance and weight, comfort, long period of service – and the rubber mixtures must contained good adhesion, uniformity and homogeneity.
These factors are dependent on the initial mixture preparing in laboratory mixer, especially fillers and their dispersion are very important factors for rubber mixture evaluation. Hence is the technology of rubber mixtures still actual and intensively development process [1, 2].
Homogeneity or inhomogeneity of rubber mixture is possibly to testing by very sensitive method – conductivity measurement by alternating current /a.c. /, which is based on principle of charge movement. For A.C. conductivity measurements was used programmable automatic RCL meter type PM 6306. We can evaluate rubber mixture by measurement of the capacity, impedance, resistance, phase angle, dissipation and quality factor by various frequencies.
On the other hand, modern imagining methods, such as atom force microscopy /AFM/, can visualise structure inhomogeneity caused by imperfection of the technological process.

2. EXPERIMENTAL PROCEDURE

There were tested three rubber samples marked as 1, 2 and 3. These were obtained from the same composition mixture, but out of different places / see Fig. 1/.

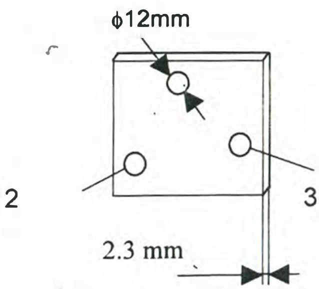

Fig. 1: Schema of sample distribution

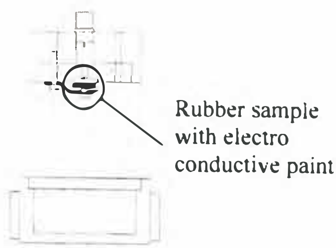

Fig. 2: Sample holder

Sample /diameter 12mm, thickness 2,3mm/ was snapped into holder /see Fig. 2/, which was connected to PC and automatic programmable RCL meter. For monitoring of presented electrical properties was created the software for PC communication at Faculty of Industrial Technologies. Samples were prepared according to the following recipe.

Tab. 1: Composition of the studied sample

Components	Dose in DSK
Sulphur	1-2
Sulfenax	1-1,5
SKD 2	20-30
BUNA	50-60
KRALEX	-
Ultrasil (SiO2)	20-40
Vulcan (carbon black)	25-45
ZnO	2,8-3,5
Dusantox	1-2
Flectol	1,5-2,5
Stearin	1,7-2,2

Tab. 2: Fillers dose

	Dose in DSK
Carbon black ratio (%)	20,5
Silica ratio (%)	13,5

The sample density for three different samples of the same composition is shown in Table 3.

Tab. 3: Values of densities of rubber samples

Sample	1	2	3
density [kg/m³]	*1204,09*	*1217,48*	*1191,64*

The experimental apparatus for measurements of a.c. electrical conductivity is schematically presented in Figure 2. A schematically view and phase graph are shown in Fig. 4.

Fig. 3: A.c. conductivity apparatus

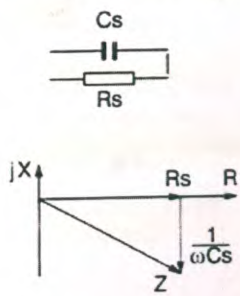

Fig. 4: serial scheme of a.c. measurement

The next was appreciated the quality preparation and homogeneity of studied samples by AFM / type NT – 206 /.

3. EXPERIMENTAL RESULTS AND DISCUSSION

We started by the analysis of electrical properties of rubber blends. On these three samples, electrical properties, such as a.c. electrical conductivity σ, electrical resistance R, capacity C, phase angle δ, quality and dissipation factor Q, D were measured. Following were calculated values of complex modulus /their real and imaginary part/, conductivity, tangent of loss angle, complex electrical modulus /their real and imaginary part/ [3, 4].

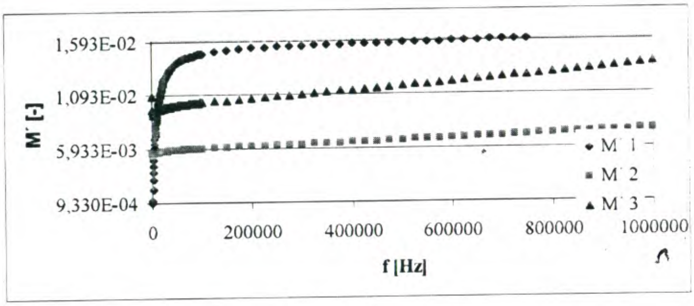

Fig. 5: Dependence of real electrical modulus versus frequency

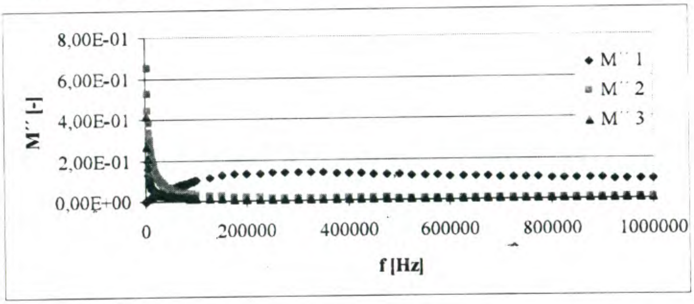

Fig. 6: Dependence of imaginary electrical modulus versus frequency

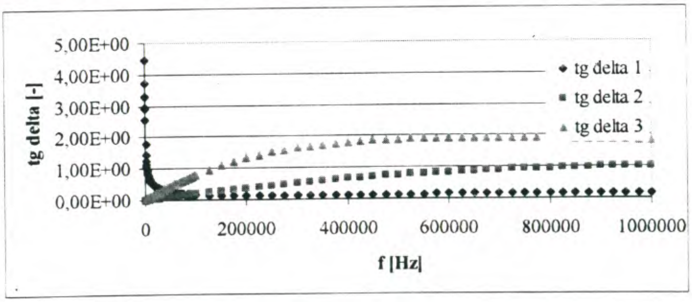

Fig. 7: Dependence of loss angle versus frequency

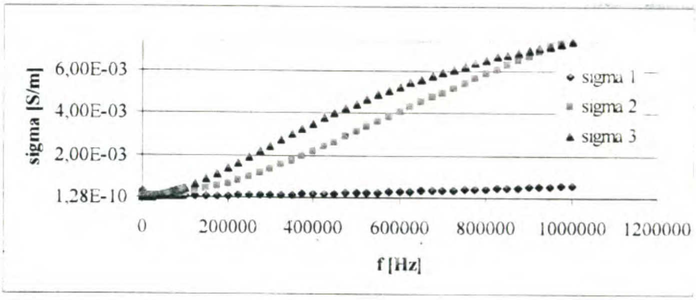

Fig. 8: Dependence of electrical conductivity versus frequency

To verify these experimental facts we studied samples also by AFM. Very attractive are inhomogeneities of rubber samples see the Fig. 9-11.

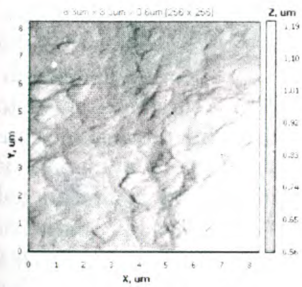

Fig. 9: AFM of sample 1

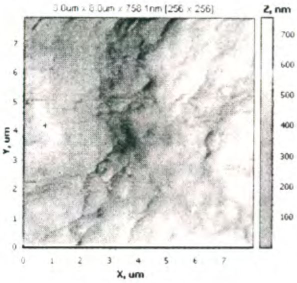

Fig. 10: AFM of sample 2

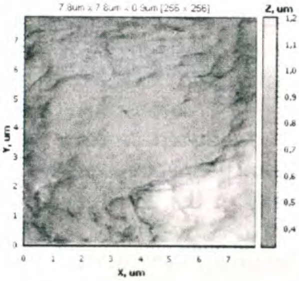

Fig. 11: AFM of sample 3

4. CONCLUSION

From the received results it is possible to say that behaviour of samples 1 and 3 has quite similar character, but mixture 2 has so much different behaviour – see the figures 5 - 8. The results enable us to conclude that different mechanisms of electrical modulus, loss angle and electrical conductivity are presented, which prove the working hypothesis about spatial inhomogeneity of prepared samples. This fact is also supported by "AFM pictures evaluation", where is seen a worse dispersion of fillers /especially carbon black/ for the samples of the same composition /see Table 1/. Sample 2 has worse preparing quality in consequence of often occurring of bright and dark places – see figure 10 / nano scale-

5. REFERENCES

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